

We also applied dual-wavelength laser radiation on human teeth. We sliced human tooth to the thin pieces with a thickness of 0.3 mm and radiated laser pulses on them. Figure 7 shows the cutting section of human tooth after laser irradiation of 5 pulses with a repetition rate of 3 Hz. When irradiated with two lasers, we obtained 15% larger depth than irradiated with Er:YAG only. On the teeth samples, we observed ablation effect that is similar to the ones for alumina balls and at the optimum condition described above, we obtained deep and sharp ablation holes on the teeth.

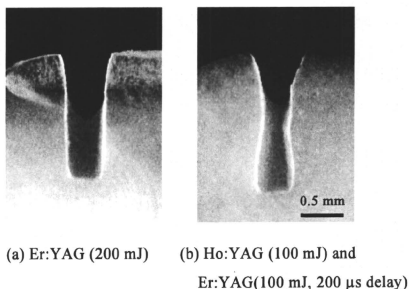


Fig. 7 Cutting section of human dentin after laser irradiation

3. Conclusion

Laser ablation experiments on hard tissues are performed by using a combined beam of Ho:YAG and Er:YAG laser light. An alumina ball is used as a hard-tissue model and ablation phenomenon are observed by an ultra-high-speed camera. The results show that the two lasers give different ablation effects due to different absorption coefficient in water contained in the tissues. When the two lasers are combined and the sample is irradiated with them, we observed that ablation capabilities are highly dependent on the delay time between two lasers and when Er:YAG laser was radiated after Ho:YAG with a delay time of 200 μ s, 40% higher depth of ablated hole is made. We suppose that the Ho:YAG pulse induces heat and this causes decrease of absorption coefficient of water. And therefore, Er:YAG laser beam can penetrates deeper from the surface.

References

1. A. Aoki, et al., "Comparison between Er:YAG laser and Conventional Technique for Root Caries Treatment in vitro," *J. Dent. Res.*, **77**, 1404-1414 (1998)
2. M. K. Yiu, et al., "Clinical Experience With Holmium:YAG Laser Lithotripsy of Ureteral Calculi," *Lasers Surg. Med.*, **19**, 103-106 (1996)
3. H. Lee, et al., "Urinary calculus fragmentation during Ho:YAG and Er:YAG lithotripsy," *Lasers Surg. Med.*, **38**, 39-51 (2006)
4. K. F. Chan, et al., "Free Electron Laser Ablation of Calculi: An Experimental Study," *IEEE J. Quantum Electron.*, **7**, 1022-1033 (2001)
5. K. L. Vodopyanov, "Saturation studies of H₂O and HDO near 3400 cm⁻¹ using intense picosecond laser pulses," *J. Chem. Phys.*, vol. **94**, no. 8, pp. 5389-5393 (1991)

Fabrication of hollow optical fiber with a vitreous film for CO₂ laser light delivery

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Abstract

Vitreous film based on the structural unit R₂SiO, where R is an organic group, is newly used as the reflection layer in the hollow optical fiber for CO₂ laser delivery. A smooth vitreous film is formed at room temperature by using the liquid-phase coating technique. The vitreous film-coated silver hollow optical fibers achieve low-loss property for lasers in the infrared regions by properly selecting fabrication conditions. A hollow fiber with thicker vitreous film designed for CO₂ laser light showed acceptable loss as an output tip. It is shown that the hollow tip is of high durability to withstand several cycles of autoclave sterilization.

Keywords: hollow fiber, CO₂ laser, infrared laser.

1. Introduction

CO₂ laser radiating at the wavelength of 10.6 μm has been used as the energy source of laser scalpel because CO₂ laser light has a high incision capacity as well as coagulation effect. Therefore CO₂ laser has become one of the widely used medical lasers in clinician. As the transmission media for infrared laser light, hollow optical fiber [1-3] is one of the commonly used infrared fibers. We have been doing research on dielectric coated metallic hollow optical fiber for various industrial and

medical lasers [4].

For the dielectric coated metallic hollow optical fiber, silver is normally used as the metallic layer, because silver has a high reflection rate in the infrared wavelength region. Cyclic olefin polymer (COP) [3] is one of the normally used dielectric materials. COP-coated silver hollow optical fibers obtained low-loss property not only in the infrared region but also in the visible region. Simultaneous delivery for infrared and pilot laser beam is possible by using this kind of hollow fiber. In medical laser treatment through hollow optical fiber, CO₂ laser irradiated the diseased tissue with the distal end of the fiber contacting the target. Therefore durability of the hollow fiber is important. Hollow optical fiber tip is often sterilized by an autoclave treatment for medical applications after irradiation. COP-coated hollow optical fiber has an obvious deterioration after the sterilization treatment. This can be caused by the somewhat weak adhesion between COP and silver layer.

In this paper, we report the inorganic material coated on silver hollow optical fiber for CO₂ laser delivery. Normally, inorganic materials have higher temperature capability and stronger adhesion to silver layer. The inorganic material is a vitreous layer that is formed by using an OC-300 [5, 6] solution. A smooth hardened film can be formed on the silver layer with the treatment of hardener solution at room temperature by using liquid-phase coating technique. The OC-300 coated hollow optical fiber shows stronger durability in medical application.

2. Inorganic coating material for hollow fiber

We tried to use an inorganic material OC-300 [5, 6] as the dielectric film. OC-300 is a semi-inorganic polymer which is originally developed as a protecting painting for outside wall of buildings or ground floor. It is a commercially available product, which is sold in a two-solution set. OC-300 is the trade name of the coating material. It is a semi-inorganic polymer based on the structural unit R₂SiO, where R is an organic group. The material is characterized by wide-range thermal stability, water repellence, and physiological inertness.

In the two-solution set, one is the main paint solution and the other is a hardener solution. When the main solution is mixed with the hardener solution, a hardened vitreous film can be formed with the catalysis of water in the air. Figure 1 shows the procedure of the vitreous film formation. The film has properties similar to that of a SiO₂ film.

In this research, the mixing rate of the main paint solution and the hardener was 10:1. The concentration of OC-300 solution is calculated by considering mixing ratio of the above solution and the paint thinner. The final solution is mixed by using a magnetic stirrer for about five minutes. And the solution is stored for a couple of hours to become stabilized before used in liquid-phase coating process. Comparing with the COP solution, the OC-300 solution has a lower viscosity.

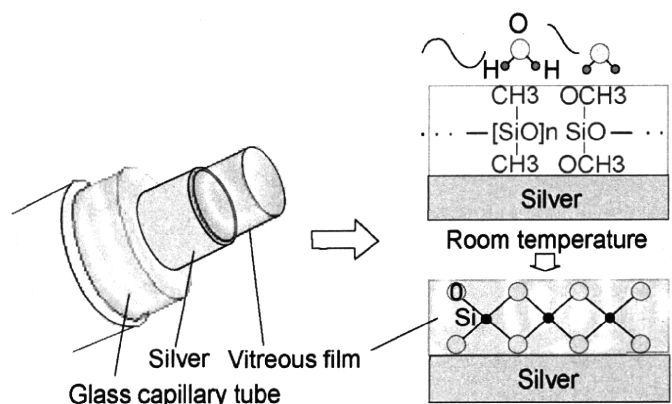


Figure 1. Structure of OC-300 coated silver hollow optical fiber

3. Fabrication and transmission property

We use chemical deposition method to plate silver layer on the glass capillary. The silver layer has a smoother surface when we pretreated the inner surface with a SnCl_2 solution. This is a guarantee for high performance hollow optical fiber. Details of the process had been described elsewhere [3].

The dielectric layer is important for a low-loss fiber. Liquid-phase coating method [7] was used to coat an OC-300 layer. The merit of using OC-300 as the dielectric layer is not only for its proper refractive index nearer to the optimum of 1.41 but also for the low temperature of film formation. This property gives great credit to a high flexible hollow fiber. As we have shown [8], the flexibility of a glass hollow fiber will deteriorate largely due to high temperature curing process, while the process is often necessary in many dielectric film-coating techniques.

Thick film coating of OC-300 layer for CO_2 laser light transmission was proved to be possible. The viscosity of the mixed solution can be modified by changing the ratio of the two solutions. As we have known that high viscosity leads to thicker film in the liquid-phase coating technique. Relationship between OC-300 film thickness and the concentration of OC-300 solution is shown in Fig. 2. The OC-300-coated silver (OC300/Ag) hollow optical fiber is 10 cm long with 1 mm inner diameter. And the flowing speed of OC-300 solution is around 1.6 cm/min in the fabrication. After the liquid-phase coating, the fiber was treated at the room temperature with nitrogen gas flowing through the hollow core for 24 hour. The liquid-phase OC300 film was changed to a vitreous layer on the silver surface. As we have known, OC-300 solution with high concentration leads to a thicker OC-300 layer. For laser light in mid-infrared region, such as CO_2 laser radiating at $10.6 \mu\text{m}$, a relatively thicker OC-300 film of about $1 \mu\text{m}$ is required. We note that the concentration of OC-300 solution is about 65 wt% in fabricating hollow optical fiber for CO_2 laser light.

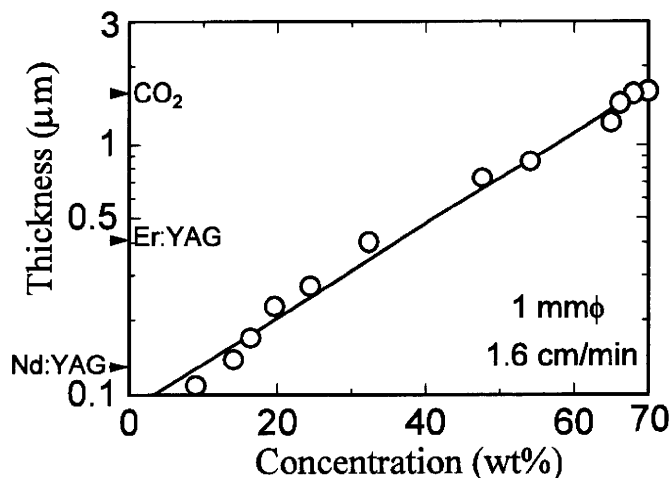


Figure 2. OC-300 film thickness as a function of the solution concentration. The flowing speed of OC-300 solution was around 1.6 cm/min and the fiber is 10 cm long with 1 mm inner diameter.

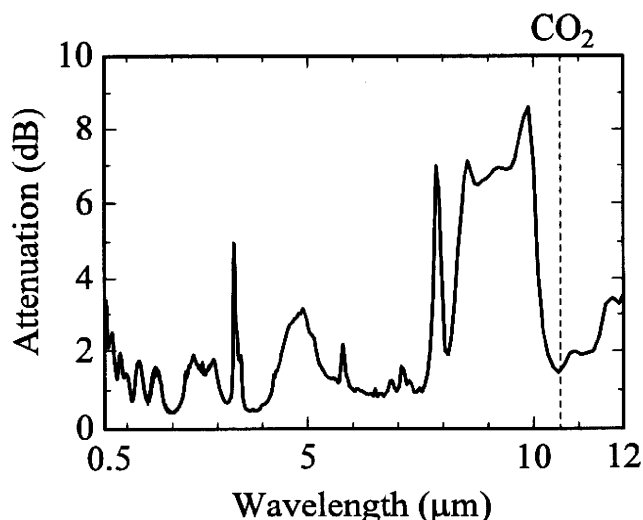


Figure 3. Loss spectrum of OC-300/Ag hollow fiber in the wavelength region from the visible to the mid-infrared. The fiber is 10 cm long with 1 mm inner diameter.

Figure 3 shows the loss spectrum of OC-300/Ag hollow fiber in the wavelength region from the visible to the mid-infrared. The fiber is 10 cm long with 1 mm inner diameter. According to the interference peaks in the visible and near infrared regions, the film thickness of the vitreous layer is calculated as 1.2 μm . The absorption peak at the wavelength of 3.5 μm is caused by impurities that contains -CH band in the main solution. The wide loss peak in the wavelength region from 8 μm to 10 μm comes from the absorption of Si-O band in the vitreous film over the silver layer. The interference

peaks in the visible and near infrared wavelength region show a smooth dielectric layer on the silver surface. The fiber attains low loss at the wavelength of 10.6 μm for CO₂ laser light.

4. Durability for sterilization treatment

Figure 4 shows the transmission property of OC-300/Ag hollow fiber for CO₂ laser light after sterilization cycles by using an autoclave device. The autoclave device is standard sterilization equipment for medical applications.

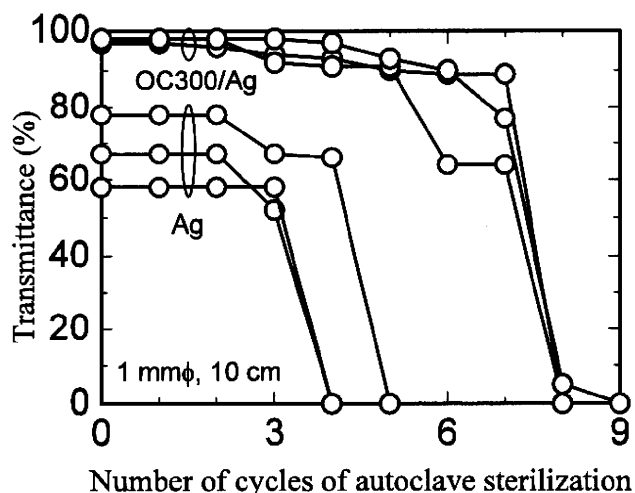


Figure 4. Transmittance for CO₂ laser light of two kinds of hollow fiber after autoclave sterilization treatment. Fibers are 10 cm long with 1 mm inner diameter.

The output of CO₂ laser used in the measurement was set as follows. The input power was 0.15 W. The treatment condition of the autoclave is 135°C for 25 minutes per cycle. The humidity and the pressure in the sample chamber of the autoclave are 100% and 2 atmospheric pressure, respectively. Transmission properties of a silver coated hollow optical fiber are also added for comparison. Data came from around three samples for each kind of hollow fiber. After each autoclave treatment cycle, samples are dried at room temperature for 30 minutes while nitrogen gas flowing through the hollow core at a lowing flowing rate of 100 ml/min. Then the next measurement and sterilization cycle was conducted. This drying process was taken to evaporate the water remained inside of the hollow core of the fiber during autoclave treatment. Without the drying process, damages occur on the dielectric layer of the hollow optical fiber when CO₂ laser light is delivered. Because water on the inner surface absorbs the CO₂ laser light energy and instant temperature is as high as several hundred degrees centigrade. Vitreous film or even the silver layer can be broken due to the high temperature.

We note in fig. 4 that Ag only hollow optical fibers have low transmittance and obvious deterioration after autoclave treatment. Temperature and humidity in the autoclave caused secession between silver layer and the glass surface of hollow optical fiber. After several treatment cycles the silver film deteriorated largely. On the other hand, OC300/Ag hollow optical fibers obtain obviously higher transmittance for CO₂ laser light and the property was stable in several treatment circles. The film thickness of the vitreous layer is 1.2 μm. For the 10 cm long tip, with an inner diameter 1 mm, its loss is less than 0.5 dB. It is acceptable for most medical device used as an output tip. This means that OC-300 adheres strongly on the silver surface and protect silver layer against humidity damage under high pressure.

5. Conclusion

We used an inorganic material as the reflection layer in hollow optical fiber for CO₂ laser light delivery. The vitreous film can be easily formed at room temperature by using liquid-phase coating method. A thicker OC-300 film with 1.2 μm thickness was successfully coated and the fiber attained low-loss property for CO₂ laser light radiating at the wavelength of 10.6 μm. The loss for the 1 mm inner diameter, 10 cm length OC300/Ag hollow fiber was 0.2 dB. The OC-300 coated hollow optical fibers are of low-loss property and high durability. It can be used in a relatively harsh industrial environment or used as a probe in medical field for repeating utilization in medical applications.

Acknowledgements

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References

1. I. Gannot, S. Schrunder, J. Dror, A. Inberg, T. Ertl, J. Tschepe, G. J. Muller, and N. Croitoru, "Flexible waveguides for Er-YAG laser radiation delivery," *IEEE Trans. Biomedical Eng.* **42**, 967-972 (1995).
2. R. K. Nubling and J. A. Harrington, "Hollow waveguide delivery system for high-power,

- industrial CO₂ lasers,” *Appl. Opt.* **35**, 372-380 (1996).
3. Y. W. Shi, Y. Wang, Y. Abe, Y. Matsuura, M. Miyagi, S. Sato, M. Taniwaki, and H. Uyama, “Cyclic olefin polymer-coated silver hollow glass waveguides for the infrared,” *Appl. Opt.* **37**, 7758-7762 (1998).
 4. M. Miyagi and S. Kawakami, “Design theory of dielectric-coated circular metallic waveguides for infrared transmission,” *IEEE J. Lightwave Technol.* **LT-2**, 116-126 (1984).
 5. K. Iwai, Y. W. Shi, M. Miyagi, X. S. Zhu, and Y. Matsuura, “Hollow infrared fiber with an inorganic inner coating layer with high durability,” *Proc. of SPIE* **6433**, 64330L-1-64330L-7 (2007).
 6. K. Iwai, M. Miyagi, Y. W. Shi, X. S. Zhu, and Y. Matsuura, “Infrared hollow fiber with a vitreous film as the dielectric inner coating layer,” *Opt. Lett.* **32**, 3420-3422 (2007).
 7. K. Iwai, Y. W. Shi, M. Miyagi, and Y. Matsuura, “Improved coating method for uniform polymer layer in infrared hollow fiber,” *Opt. & Laser Technol.* **39**, 1528-1531 (2007).
 8. Y. W. Shi, K. Ito, L. Ma, T. Yoshida, Y. Matsuura, and M. Miyagi, “Fabrication of a polymer-coated silver hollow optical fiber with high performance,” *Appl. Opt.* **45**, 6736-6740 (2006).

Transmission properties of dielectric-coated hollow optical fibers based on silver-cladding-stainless pipe

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Abstract

Silver-cladding-stainless pipe is used as the supporting tube for the infrared hollow fiber to obtain high durability and strong mechanical strength. For the dielectric inner-coating layer, cyclic olefin polymer (COP) and silver iodide (AgI) are used to lower the transmission loss. The COP layer is formed by using liquid-phase coating method as it is done before. For the AgI layer, liquid-filling technique is developed to reduce the waste liquid of iodine solution. Rigid hollow fiber with optimized COP or AgI inner film thicknesses for CO₂ laser light were fabricated and reasonable transmission losses for an output tip was demonstrated.

KEYWORD: hollow fiber, output tip, infrared laser.

1. Introduction

The CO₂ laser radiation at the wavelength of 10.6 μm has found applications in industrial processing owing to its high output power. It is also one of the most commonly used lasers in medical field for laser surgery because of the high water absorption coefficient at the wavelength. Laser power

transmission system has been critically required and actively developed [1-6] because of the wide application of CO₂ laser. We have developed hollow fiber with polymer and silver inner-coating films based on glass capillary with high flexibility. Low-loss infrared hollow fibers with inner diameters from 320 μm to 1000 μm and length of 2 meters were successfully developed [6].

In medical application, such as dentistry and otorhinolaryngology, sterilization process must be done for the recycled output probe of infrared laser delivery system. It has been shown that the dielectric-coated silver hollow tips were damaged after several sterilization cycles [7] by using autoclave. It was observed that the coated silver layer was detached from the inner surface of the glass capillary. This is mainly caused by the rather thin silver film. In order to obtain a smooth silver surface for a better optical film, the silver film had to be rather thin [8], which is normally from 50 nm to 200 nm thick.

In this paper, we propose to use metal supporting pipes for the fabrication of the infrared hollow fiber. Cyclic olefin polymer (COP) [9] and AgI [10] were used as the dielectric coating material. COP is non-toxic, transparent material in the infrared regions, and capable of forming optical film with high quality. AgI is a traditional infrared material, which suits high power delivery for its good adhesion with silver film and heat-resistance property. These two kinds of dielectric film were used in the hollow output probe in the fabrication and transmission properties were experimentally evaluated.

2. Supporting pipe

We have used a glass capillary as a supporting tube for the hollow fiber because of its smooth inner surface. To increase the heat resistance and durability for sterilization process, we fabricate some hollow fibers using several kinds of metal tubes instead of the glass capillary. They are a stainless steel pipe (SUS) with a smooth polished inner surface, a gold-coated stainless steel pipe (Au/SUS), and a silver-clad stainless steel pipe (Ag/SUS) [11]. Figure 1 shows the structure of various metal pipes. The inner polished SUS pipe is commercially available.

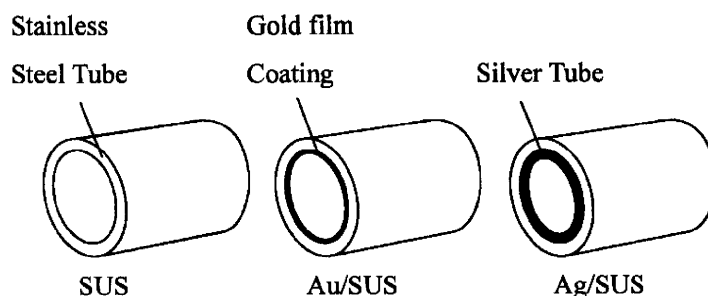


Figure 1. Structure of various metal pipes

The gold thin film of Au/SUS pipe was coated by electric plating on the inner surface of the SUS pipe. Ag/SUS pipe was integrally formed as a metal pipe made of a silver-clad layer and a stainless steel layer by extruding a cylindrical silver pipe arranged inside to a stainless steel pipe arranged outside. After forming the pressure-bonding silver and stainless steel dual-clad pipe, the inside wall of the silver-clad layer was polished to a mirror-smooth state. The silver clad layer has the wall thickness of 100 μm and expected to be of high durability and low loss when inner-coated with optical films.

Figure 2 shows transmission loss spectra for several kinds of pipes from the visible to the mid-infrared regions. We estimated some metal pipes shown in Fig. 1 and a silver-plated silica capillary (Ag/SiO₂) as well for comparison.

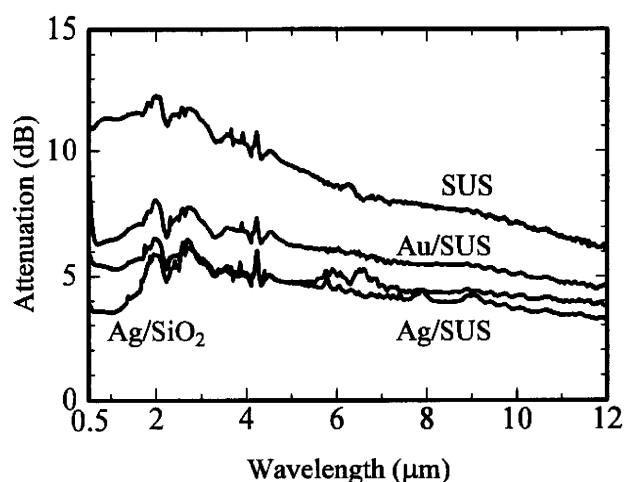


Figure 2. Loss spectra of metal pipes and Ag-plated silica capillary from visible to infrared regions.

Table 1 summarizes the transmission losses for CO₂ laser light through these pipes as well as their sizes. For measurement of the loss spectra in Fig 2, we used a white light source and an optical spectral analyzer up to 1.7 μm in wavelength and a NiCr heat source and an infrared monochromator from 1.7 μm to 12 μm in wavelength.

Table 1. Sizes and losses of CO₂ laser light through various kinds of metal pipes and Ag-plated silica capillary

Material	Size (mm)			Loss (dB)
	ID	OD	L	
SUS	0.94	1.2	285	3.7
Au/SUS	0.94	1.2	285	2.1
Ag/SUS	0.75	1.2	280	1.7
Ag/SiO ₂	0.75	0.9	280	1.8

ID: Inner Diameter, OD: Outer Diameter, L: Length

In Table 1, the transmission losses for CO₂ laser light shown were estimated by the ratio of input power launched through an incident lens to output power at the distal end. In general, transmission losses for CO₂ laser light are lower than loss spectra at 10.6 μm in wavelength in Fig.2, because the laser source can excite lowest order mode (HE₁₁ mode) more efficiently than the incoherent light source. Although we have not measured quantitatively the inner roughness of each pipe, we can infer that the inner roughness of Ag/SiO₂ is smaller than other metal pipes, because Ag/SiO₂ has a rather low-loss in the visible and near-infrared regions as shown in Fig.2. The glass capillary has a very smooth inner surface that guarantees smooth silver layer plating and thus causes smaller additional loss. The longer wavelength is, the less the additional loss due to inner surface roughness increases. As a result, the transmission property of the Ag/SUS is equivalent to that of the Ag/SiO₂ in the mid-infrared region.

We made the inner polished Ag/SUS pipe up to 1 m long, which is restricted by the polishing machine. Although it is difficult to obtain longer Ag/SUS pipe, the metal pipe has strong mechanical strength, heat resistance and capability of withstanding sharp bending. Therefore it is proper and safe for the application as an output probe attached at the distal end of laser delivery system. On the other hand, the hollow fiber using a glass capillary is suitable for long transmission of laser light because of its flexibility and inner smooth surface.

3. Polymer inner coating

Optical film coating on the metal surface can dramatically increase the reflectance of the surface. Therefore, it is an ordinary method to coat a dielectric film on the inner surface of the metal pipe to reduce the transmission loss. Cyclic olefin polymer (COP) and silver iodide (AgI) are two successful dielectric coating materials for infrared hollow fiber. For a target wavelength, there is an optimum film thickness [12] for each dielectric material.

Figure 3 shows the theoretical loss of the HE₁₁ mode in the dielectric coated hollow fiber as a function of dielectric film thickness. The target wavelength is 10.6 μm for CO₂ laser light. In the calculation, the complex refractive index for Ag at the wavelength of 10.6 μm is 13.5 - j 75.3. The refractive indices for COP and AgI are 1.53 and 1.95 [13], respectively. We note in Fig. 3 that optimum thicknesses for minimum loss are 1.36 μm and 0.98 μm for COP and AgI layer, respectively. The loss of COP/Ag hollow fiber is a little smaller than that of the AgI/Ag hollow fiber because the optimum refractive index for the dielectric layer is theoretically 1.41.

We used the COP as the dielectric coating material. The COP polymer was dissolved in cyclohexane. The solution was forced to flow through the metal pipe and a liquid-phase film was formed on the inner surface. After the curing process, the film was solidified to form a stable polymer

layer. Detailed fabrication techniques and parameters to control film thickness have been published elsewhere [14].

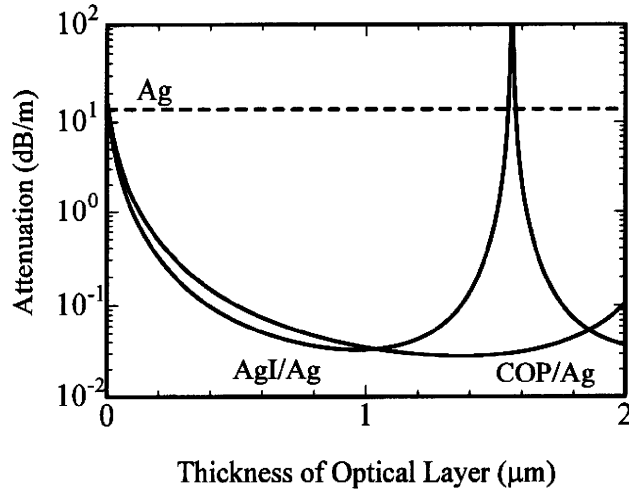


Figure 3. Theoretical losses for the HE₁₁ mode in the hollow fiber of 0.75 mm inner diameter as a function of dielectric film thickness for CO₂ laser light.

Fig.4 and Table 2 show loss spectra and losses for CO₂ laser light of several kinds of COP-coated hollow fibers, respectively. The COP layer was coated on the inside wall of each metal pipe or silver-plated silica capillary in Table 1.

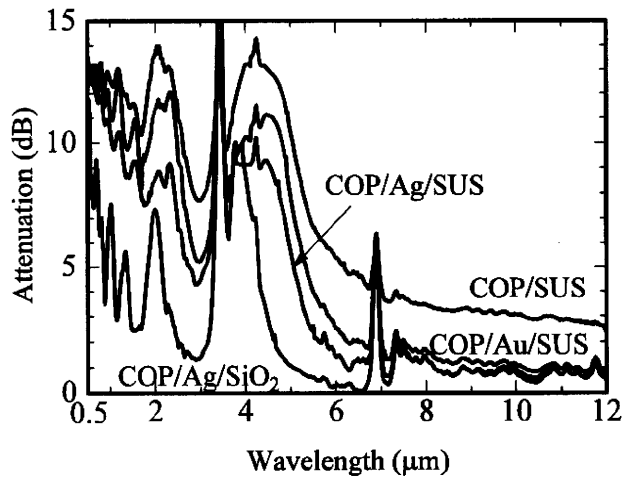


Figure 4. Loss spectra of COP-coated metal pipes and COP-coated Ag-plated silica capillary

In Fig. 4, loss peaks in the wavelength region shorter than 5 μm came from the interference effect of light in the thin COP optical film. According to the position of the interference peaks, the film thicknesses for the COP layer can be calculated. For the four kinds of COP-coated metal pipes,

the film thickness was estimated to be range from 0.85 μm to 0.95 μm . Comparing with the theoretical optimum COP thickness of 1.36 μm shown in Fig.3, the practical thickness was made somewhat thinner. We take into account that the bottom loss range is broad around the optimum thickness and the thicker film is likely to cause the rougher inner surface. Therefore, just a little thinner film than the theoretical optimum thickness is normally preferred in fabrication. As shown in Table 2, low loss under 0.2 dB for CO₂ laser light was obtained for COP-coated Ag hollow pipes.

Table 2. Sizes and losses of CO₂ laser light through various kinds of COP-coated metal pipes and COP-coated Ag-plated silica capillary

Material	Size (mm)			Loss (dB)
	ID	OD	L	
COP/SUS	0.94	1.2	285	0.6
COP/Au/SUS	0.94	1.2	285	0.22
COP/Ag/SUS	0.75	1.2	280	0.18
COP/Ag/SiO ₂	0.75	0.9	280	0.13

ID: Inner Diameter, OD: Outer Diameter, L: Length

4. Silver iodide coating

The coating technique for the AgI layer is an iodination process that changes part of the silver layer into AgI layer. The iodine cyclohexane solution is flown through the silver coated tube and the upper surface of the silver layer is changed into silver iodide. This AgI coating technique has been working well for hollow fiber with inner diameters ranging from 0.25 to 0.7 mm [15]. However, a rather high flowing rate is necessary in the iodination process. To form the AgI layer, 20 ml iodine solution was needed. We propose a new method that significantly reduces the waste of the iodine solution.

A syringe is used to fill the iodine solution into the Ag/SUS pipe. The pipe is fully filled and the solution is kept for a period of time for AgI layer formation. Then the iodine solution is pushed out. By using the newly developed method, the volume of the iodine solution is only 0.1 ml for the short metal pipe with 0.75 mm bore diameter and 280 mm long. In the fabrication process, the iodine solution was made by dissolving the iodine pellet in cyclohexane. And then the solution was stirring for fully mixture in a supersonic device for ten minutes.

Figure 5 shows the AgI film thickness versus liquid-filling time. The concentrations of the iodine solution are also shown. The dashed line shows the theoretical optimum film thickness for Er:YAG laser that irradiates at 2.94 μm . The fabrication parameter for Er:YAG hollow pipe could be like 60

seconds filling time for iodine solution with 0.5% concentration. The transmission loss for Er:YAG laser light through the AgI/Ag/SUS pipe with 0.25 μm thick AgI film was 0.4 dB (92 %). Film thickness tends to be thicker when the filling time is prolonged. However, there exists a saturation time for a certain concentration as it can be observed in Fig. 5. This is because the concentration of Iodine solution decreases with the chemical reaction time and the AgI formed on the surface also restrains the chemical reaction speed.

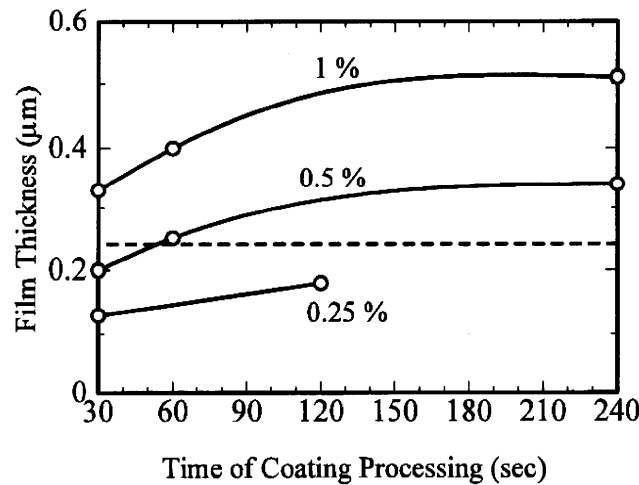


Figure 5. AgI film thicknesses versus liquid-filling time for solutions with various concentrations

To form a thicker AgI film for CO₂ laser light, we used 1 % concentration solution and multiple liquid-filling processes. As one process could not achieve enough film thickness, we repeat the same filling process three to five times. Figure 6 shows the AgI film thickness versus repeating times. Holding times for each filling process are also shown in this figure.

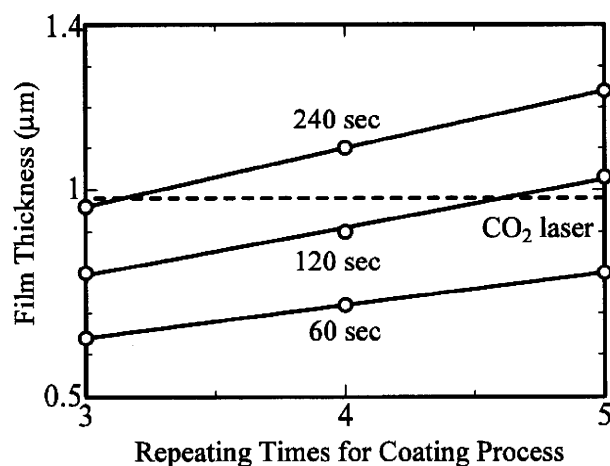


Figure 6. AgI film thicknesses versus repeating times for liquid-filling process.

The dashed line shows the theoretical optimum film thickness for CO₂ laser light that irradiates at 10.6 μm. It is seen that we need to repeat the process 3 repeat times for 240 seconds filling time, or 4 repeat times for 120 seconds filling time. As the 240 second filling time is longer per filling process, the concentration of the iodine solution decreased largely and the surface of the AgI film tends to be rough. So we selected 120 seconds filling time and 4 times repeat processes for fabricating conditions of CO₂ laser light hollow fibers.

Figure 7 shows the loss spectra of the AgI/Ag/SUS pipes fabricated with 2 to 4 liquid-filling times. As the repetition times of liquid-filling increases, the inner AgI layer becomes thicker and the interference peaks shift to longer wavelength. The film thickness for 2, 3, and 4 repetition times are estimated to be 0.65 μm, 0.78 μm, and 0.89 μm, respectively. One time filling process approximately increases the film thickness by 0.1 μm.

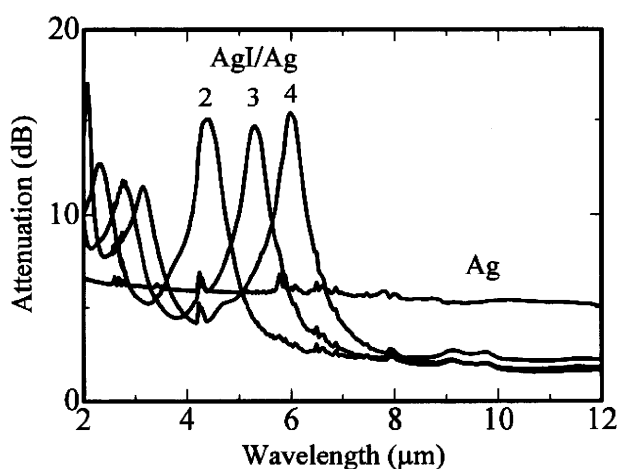


Figure 7. Loss spectra of AgI/Ag/SUS pipes using multiple iodination processes. The numbers in the figure represent iodination process times.

The AgI film thickness can be controlled by adjusting the holding time and repetition times of Iodine solution filling. On the other hand, the AgI film can be etched away by sodium thiosulfate solution. Figure 8 shows the variation of AgI film thickness with the treatment of sodium thiosulfate water solution. The original AgI/Ag hollow fiber has an interference peak at the wavelength of 6.4 μm, which corresponds to the AgI film thickness of 0.95 μm. Spectra A, B and C were measured after a series of decoating processes on the original AgI/Ag/SUS pipe. Spectrum A was the first measurement result after the sodium thiosulfate water solution treatment, where the solution was flown through the pipe for 30 seconds with speed of 3 cm/second and the concentration of sodium thiosulfate was 0.1 ml / L. Spectrum B was the second result, which is added to the sample with spectrum A under conditions

of 300 second flow time and the concentration of 0.05 ml / L. Spectrum C was the third result after further additional treatment under conditions of 180 second solution flow time and the concentration of 0.1 ml / L. The AgI film thickness for A, B, and C were estimated to be 0.81, 0.8, and 0.66 μm , respectively.

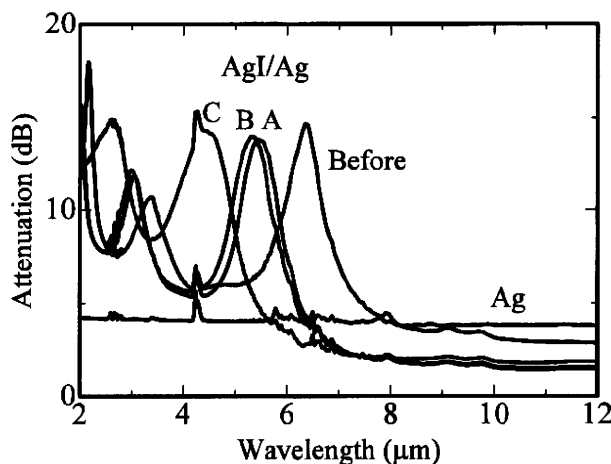


Figure 8. Loss spectra of AgI/Ag/SUS pipe treated with sodium thiosulfate decoating process.

According to the spectrum change from A to B, the sodium thiosulfate concentration of 0.05 ml / L is so weak that the AgI film etching rate is very little. The film thickness variation from original to A and from B to C was the same as 0.14 μm , while the treatment time was 30 and 180 seconds respectively. In this case, the flowing time longer than 30 second is not effective, although the sodium thiosulfate concentration is enough for AgI etching process. In this way, we can mend the AgI/Ag hollow fiber with too thick AgI layer.

Figure 9 shows the loss spectra for AgI/Ag/SUS pipes with different AgI film thicknesses. The film thicknesses for the three pipes are 0.81, 1.01, and 1.22 μm , respectively. Sharp interference peaks showed that uniform and smooth AgI films were formed on the inner surface of the Ag/SUS pipe. According to the theoretical calculation, the pipe with 0.98 μm thick AgI film should has lowest loss at 10.6 μm in wavelength. As shown in Fig. 9, the loss for the AgI/Ag pipe with 0.81 μm AgI film is as low as that of AgI/Ag with 1.01 μm that is closer to the optimum thickness of 0.98 μm . This is because the bottom loss range is broad around the optimum thickness and the thicker film that needs longer iodination time or more repetition processes is likely to cause non-uniformity of AgI layer.

Transmission loss for CO₂ laser light though the AgI/Ag/SUS pipe is shown in Table 3, which was equal to that of COP/Ag/SUS. As compared to the loss of 1.7 dB for the Ag/SUS pipe, AgI or COP film coating on the Ag layer is much effective in reduction the transmission loss.

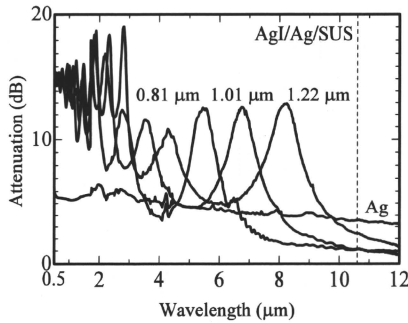


Figure 9. Loss spectra for AgI/Ag/SUS pipes with various AgI film thicknesses.

Table 3. Size and loss of CO₂ laser light through AgI-coated Ag pipe

Material	Size (mm)			Loss (dB)
	ID	OD	L	
AgI/Ag/SUS	0.75	1.2	280	0.18

ID: Inner Diameter, OD: Outer Diameter, L: Length

The hollow fiber using the metal pipe as the base material is not flexible but mechanically strong and does not break even under small radius bending. We fabricated a bent AgI/Ag/SUS pipe, which is 23 cm long and bent at 90 degrees with 4-cm bending radius as shown in Fig.10. The transmission loss for CO₂ laser light through this bent AgI/Ag/SUS was 0.7 dB (85 %), which is a reasonable loss for an output probe of an infrared laser power delivery system.

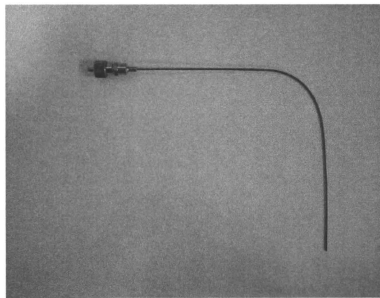


Figure 10. Photograph of bent AgI/Ag/SUS pipes with FC connector

5. Conclusion

In order to obtain strong mechanical strength for the probe of infrared power delivery system, we fabricated short infrared hollow pipe based on various metal tubes. The tubes are stainless steel pipe (SUS), gold-coated stainless steel pipe (Au/SUS), and silver-clad stainless steel pipe (Ag/SUS). It is shown that fiber with inner optical film coating based on Ag/SUS is of the lowest loss property. We have developed liquid-filling method to minimize waste and multiple iodination process to form a thicker film. A decoating process was also proposed to accurately control the AgI film thickness. Loss for the COP or AgI coated Ag/SUS hollow pipes is smaller than 0.2 dB. The tube is with a length of 280 mm and an inner diameter of 0.75 mm. The Ag/SUS tube is of strong mechanical property and low-loss property. It is proper to be used as the output probe for the long flexible infrared laser power delivery system.

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Reference

1. M. Alaluf, J. Dror, R. Dahan, and N. Croitoru, "Plastic hollow fibers as a selective infrared radiation transmitting medium," *J. Appl. Phys.* **72**, 3878-3883 (1992).
2. T. Abel, J. Hirsch, and J. A. Harrington, "Hollow glass waveguides for broadband infrared transmission," *Opt. Lett.* **19**, 1034-1036 (1994).
3. I. Gannot, S. Schründer, J. Dror, A. Inberg, T. Ertl, J. Tschepe, and G. J. Müller, "Flexible Waveguides for Er-YAG Laser Radiation Delivery," *IEEE Transactions on Biomedical Engineering* **42**, 967-972 (1995).
4. Y. Wang, A. Hongo, Y. Kato, T. Shimomura, D. Miura, and M. Miyagi, "Thickness and uniformity of fluorocarbon polymer film dynamically coated inside silver hollow glass waveguides," *Appl. Opt.* **36**, 2886-2892 (1997).
5. Y. Matsuura, A. Tsuchiuchi, H. Noguchi, and M. Miyagi, "Hollow fiber optics with improved durability for high-peak-power pulses of Q-switched Nd:YAG lasers," *Appl. Opt.* **46**, 1279-1282 (2007).

6. Y. W. Shi, Y. Wang, Y. Abe, Y. Matsuura, M. Miyagi, S. Sato, M. Taniwaki, and H. Uyama, "Cyclic Olefin Polymer-Coated Silver Hollow Glass Waveguides for the Infrared," *Appl. Opt.* **37**, 7758-7762 (1998).
7. K. Iwai, M. Miyagi, Y. W. Shi, X. S. Zhu, and Y. Matsuura, "Infrared hollow fiber with a vitreous film as the dielectric inner coating layer," *Opt. Lett.* **32**, 3420-3422 (2007).
8. C. D. Rabbii, D. J. Gibson, and J. A. Harrington, "Processing and Characterization of Silver Films Used to Fabricate Hollow Glass Waveguides," *Appl. Opt.* **38**, 4486-4493 (1999).
9. Y. W. Shi, K. Ito, Y. Matsuura, and M. Miyagi, "Multiwavelength laser light transmission of hollow optical fiber from the visible to the mid-infrared," *Opt. Lett.* **30**, 2867-2869 (2005).
10. R. George and J. A. Harrington, "Infrared transmissive, hollow plastic waveguides with inner Ag-AgI coatings," *Appl. Opt.* **44**, 6449-6455 (2005).
11. A. Hongo, H. Takamiya, and T. Koike, "Hollow fiber using silver-clad stainless steel pipe with inner dielectric layer for CO₂ laser light transmission," *Chinese Opt. Lett.* **5**, Supplement, S70-S72 (2007).
12. M. Miyagi and S. Kawakami, "Design theory of dielectric-coated circular metallic waveguides for infrared transmission," *J. Lightwave Technol.* **LT-2**, 116-126 (1984).
13. R. George and J. A. Harrington, "Cu/CuI-coated hollow glass waveguides for delivery of infrared radiation," *Opt. Eng.* **45**, 055004-1-055004-4 (2006).
14. Y. W. Shi, K. Ito, L. Ma, T. Yoshida, Y. Matsuura, and M. Miyagi, "Fabrication of a polymer-coated silver hollow optical fiber with high performance," *Appl. Opt.* **45**, 6736-6740 (2006).
15. Y. Matsuura, T. Abel, and J. A. Harrington, "Optical properties of small-bore hollow glass waveguides," *Appl. Opt.* **34**, 6842-6847 (1995).